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दुग्ध एवं दुग्ध उत्पाद — नाईट्रेट एवं नाईट्राइट का अंश निर्धारण

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(दूसरा पुनरीक्षण)

Indian Standard

MILK AND MILK PRODUCTS — DETERMINATION OF NITRATE AND NITRITE CONTENTS

PART 2 METHOD USING SEGMENTED FLOW ANALYSIS (ROUTINE METHOD)

(Second Revision)

ICS 67.100.01

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

April 2008 **Price Group 7**

Dairy Products and Equipment Sectional Committee, FAD 19

NATIONAL FOREWORD

This Indian Standard (Part 2) (Second Revision) which is identical with ISO 14673-2 : 2004 'Milk and milk products — Determination of nitrate and nitrite contents: Part 2 Method using segmented flow analysis (Routine method)' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dairy Products and Equipment Sectional Committee and approval of the Food and Agriculture Division Council.

This standard was first published in 1981 based on the earlier version of International Standard, ISO 4099 : 1978 and subsequently revised in 1997 to align with the revised ISO 4099 : 1984 under dual numbering and its scope covered determination of nitrate and nitrite contents in cheese using cadmium reduction and spectrometry. Subsequently ISO 4099 was withdrawn and revised into three parts of ISO 14673 to include in its scope a variety of milk and milk products and also including two more methods for determination of nitrate and nitrite contents.

Subsequently to align with ISO 14673 on the subject, IS 9967 is now being revised into following three parts under the general title: 'Milk and milk products — Determination of nitrate and nitrite contents'.

Part 1	Method using cadmium reduction and spectrometry and is identical to ISO 14673-1 : 2004;
Part 2	Method using segmented flow analysis (routine method) and is identical to ISO 14673-2 : 2004; and
Part 3	Method using cadmium reduction and flow injection analysis with in-line dialysis (routine method) and is identical to ISO 14673-3 : 2004.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respected places are given below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 648 : 1977 Laboratory glassware — One-mark pipettes	IS 1117 : 1975 Specification for one-mark pipettes (*first revision*)	Technically Equivalent
ISO 835-1 : 1981 Laboratory glassware — Graduated pipettes — Part 1: General requirements	IS 4162 (Part 1) : 1985 Specification for graduated pipettes: Part 1 General requirements (*first revision*)	do
ISO 1042 : 1998 Laboratory glassware — One-mark volumetric flasks	IS 915 : 1975 Specification for one-mark volumetric flasks	do

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

MILK AND MILK PRODUCTS — DETERMINATION OF NITRATE AND NITRITE CONTENTS

PART 2 METHOD USING SEGMENTED FLOW ANALYSIS (ROUTINE METHOD)

(Second Revision)

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 14673|IDF 189 specifies a routine method for the determination of the nitrate and nitrite contents of milk and milk products by segmented flow analysis. The method is applicable to milk, cheese, and liquid and dried milk products and infant foods.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 835-1, *Laboratory glassware — Graduated pipettes — Part 1: General requirements*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
nitrate content
mass fraction of nitrate determined by the procedure specified in this part of ISO 14673|IDF 189

NOTE The nitrate content is expressed as the mass in milligrams of nitrate ions (NO_3^-) per kilogram of product.

3.2
nitrite content
mass fraction of nitrite determined by the procedure specified in this part of ISO 14673|IDF 189

NOTE The nitrite content is expressed as the mass in milligrams of nitrite ions (NO_2^-) per kilogram of product.

4 Principle

4.1 Nitrate determination

A test portion is suspended in water. Part of the suspension is transferred to the analyser for dialysis. The nitrate ions are reduced to nitrite. The nitrite content is determined by a spectrometric method.

Standard nitrate solutions are determined by the same procedure. The nitrate content is calculated by comparing the reading obtained from the test portion with the readings from the standard solutions.

NOTE Any nitrite present is determined as nitrate. The amount of nitrite is generally small compared to the amount of nitrate. Infant food based on soy-proteins can be an exception to the rule. A correction for the nitrite present can be applied after determination of the nitrite content.

4.2 Nitrite determination

A test portion is suspended in a solution of ammonium and sodium chloride. Part of the suspension is transferred to the analyser for dialysis. The nitrite content is determined by a spectrometric method.

Standard nitrite solutions are determined by the same procedure. The nitrite content is calculated by comparing the reading obtained from the test portion with that from the standard solution.

5 Reagents

Use only reagents of recognized analytical grade unless otherwise specified.

5.1 Water, distilled or deionized, or water of equivalent purity, free from nitrate and nitrite ions.

To avoid the possible inclusion of small gas bubbles in the copperized cadmium column (6.9), freshly boil the distilled or deionized water and cool to room temperature. Use the thus-prepared water for the preparation of the column (9.1), to check the reducing capacity of the column (9.2), and for regeneration of the column.

5.2 Cadmium granules, of diameter 0,3 mm to 0,8 mm.

Prepare cadmium granules, if not available commercially, as follows.

Place a suitable number of zinc rods in a beaker. Cover the rods with cadmium sulfate solution (5.3). Scrape the cadmium sponge from the rods from time to time over a period of 24 h. Remove the zinc rods and decant the liquid until only sufficient remains to cover the cadmium sponge. Wash the sponge two or three times with water. Transfer the cadmium sponge to a laboratory blender together with 400 ml of the dilute hydrochloric acid (5.6) and blend for a few seconds to obtain granules of the required size. Return the contents of the blender to the beaker and leave to stand for several hours, while stirring occasionally to remove bubbles. Decant most of the liquid and immediately copperize the granules as described in 9.1.2.

WARNING — Because of its toxicity, the used cadmium should be delivered as chemical waste to the relevant authorities.

5.3 Cadmium sulfate solution, $c(CdSO_4 \cdot 8H_2O) = 40$ g/l.

Dissolve 40 g of cadmium sulfate in water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.4 Copper(II) sulfate solution, $c(CuSO_4 \cdot 5H_2O) = 20$ g/l.

Dissolve 2 g of copper(II) sulfate in water in a 100 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.5 Hydrochloric acid (HCl), ($\rho_{20} = 1,19$ g/ml).

5.6 Dilute hydrochloric acid, $c(HCl) \approx 1$ mol/l

Carefully add 80 ml of hydrochloric acid (5.5) to about 700 ml water in a 1 000 ml volumetric flask (6.4) while regularly swirling the contents. Cool the contents to room temperature. Dilute to the mark with water and mix carefully.

5.7 Disodium ethylenediaminetetraacetate dihydrate (EDTA) solution ($Na_2C_{10}H_{14}N_2O_8 \cdot 2H_2O$)

Dissolve 33,5 g of EDTA in about 900 ml water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.8 Poly(oxyethylene lauryl ether) solution, 30 % mass fraction.

5.9 Ammonium and sodium chloride solution I

Dissolve 40 g of ammonium chloride (NH_4Cl) and 200 g of sodium chloride (NaCl) in about 950 ml water in a 1 000 ml volumetric flask (6.4). Use concentrated ammonia to adjust the pH to 8,5. Dilute to the mark with water. Add 2 ml of poly(oxyethylene lauryl ether) solution (5.8) and mix.

5.10 Ammonium and sodium chloride solution II

Dissolve 10 g of ammonium chloride (NH_4Cl) and 50 g of sodium chloride (NaCl) in 950 ml water in a 1 000 ml volumetric flask (6.4). Add 20 ml EDTA solution (5.7) and mix. Use concentrated ammonia to adjust the pH to 8,5. Dilute to the mark with water. Add 2 ml of poly(oxyethylene lauryl ether) solution (5.8) and mix.

5.11 Ammonium and sodium chloride solution III

Dissolve 10 g of ammonium chloride (NH_4Cl) and 50 g of sodium chloride (NaCl) in 950 ml water in a 1 000 ml volumetric flask (6.4). Use concentrated ammonia to adjust the pH to 8,5. Dilute the contents to the 1 000 ml mark with water. Add 2 ml of poly(oxyethylene lauryl ether) solution (5.8) and mix.

5.12 Phosphoric acid solution (H_3PO_4), 85 %, ($\rho_{20} = 1{,}71$ g/ml)

5.13 Colour reagent.

Add 100 ml of phosphoric acid solution (5.12) to 800 ml water in a 1 000 ml volumetric flask (6.4). Add in the following order: 10 g sulfanilamide ($NH_2C_6H_4SO_2NH_2$) and 0,5 g *N*-1-naphthylethylene diamine dihydrochloride ($C_{10}H_7NHCH_2CH_2NH_2 \cdot 2HCl$) and mix. Dilute to the mark with water. Add 0,5 ml of poly(oxyethylene lauryl ether) solution (5.8) and mix again.

If stored in a refrigerator, the colour reagent solution may be kept for up to 1 month.

5.14 Sodium nitrate stock solution ($NaNO_3$), $c(NO_3^-) = 0{,}400$ g/l

Dry an amount of sodium nitrate in the oven (6.18) set at 110 °C to 120 °C for 2 h. Dissolve 0,548 4 g of the dry sodium nitrate in water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.15 Sodium nitrate working solution, $c(NO_3^-) = 40$ µg/ml.

Pipette (6.5) 10 ml of the sodium nitrate stock solution (5.14) into a 100 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.16 Sodium nitrate calibration solutions.

Prepare a series of sodium nitrate calibration solutions with an increasing concentration of nitrate by pipetting into six 200 ml volumetric flasks respectively, 1 ml, 2 ml, 3 ml, 6 ml, 9 ml and 12 ml of the sodium nitrate working solution (5.15). Dilute each solution to the mark with water and mix. The nitrate (NO_3^-) contents of the obtained sodium nitrate calibration solutions are 0,2 µg/ml, 0,4 µg/ml, 0,6 µg/ml, 1,2 µg/ml, 1,8 µg/ml and 2,4 µg/ml respectively.

5.17 Sodium nitrite stock solutions ($NaNO_2$).

Dry an amount of sodium nitrite in the oven (6.18) set at 110 °C to 120 °C for 2 h.

5.17.1 Sodium nitrite stock solutions I, $c(NO_2^-)$ = 0,297 mg/ml.

Dissolve 0,445 g of the dry sodium nitrite (5.17) in water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix. Stock solution I may be stored for up to 1 day.

5.17.2 Sodium nitrite stock solutions II, $c(NO_2^-)$ = 1,001 mg/ml.

Dissolve 1,502 g of sodium nitrite (5.17) in water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix. If stored in a refrigerator, stock solution II may be kept for up to 1 month.

5.18 Sodium nitrite working solutions.

5.18.1 Sodium nitrite working solution I, $c(NO_2^-)$ = 0,89 µg/ml.

Transfer with a pipette (6.5) 3 ml of the sodium nitrite stock solution I (5.17.1) to a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.18.2 Sodium nitrite working solution II, $c(NO_2^-)$ = 0,100 1 µg/ml.

Pipette (6.5) 10 ml of the sodium nitrite stock solution II (5.17.2) into a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix. Pipette 10 ml of this solution into another 1 000 ml volumetric flask (6.4). Dilute to the mark with ammonium and sodium chloride solution III (5.11) and mix.

5.19 Sodium nitrite calibration solutions.

Prepare a series of sodium nitrite calibration solutions with an increasing concentration of nitrite by pipetting into five 200 ml volumetric flasks (6.4) respectively, 10 ml, 20 ml, 40 ml, 100 ml and 200 ml of sodium nitrite working solution II (5.18.2). Except for the 200 ml solution, dilute each solution to the 200 ml mark with ammonium and sodium chloride solution III (5.11) and mix. The sodium nitrite calibration solutions have a nitrite (NO_2^-) content of 0,005 µg/ml, 0,010 µg/ml, 0,020 µg/ml, 0,050 µg/ml and 0,100 µg/ml, respectively.

5.20 Alkaline detergent.

Use one of the solutions mentioned below.

5.20.1 Extran solution, 5 % volume fraction.

Pipette (6.5) 50 ml of extran solution into a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.20.2 Sodium hydroxide solution, $c(NaOH)$ = 0,1 mol/l.

Dissolve 4 g of sodium hydroxide (NaOH) in water in a 1 000 ml volumetric flask (6.4). Dilute to the mark with water and mix.

5.21 Regenerating fluid.

Add 50 ml of EDTA solution (5.7) and 2,0 ml of dilute hydrochloric acid (5.6) to 1 litre of water (5.1) and mix.

5.22 Reference test sample.

Use as a reference test sample, samples from a milk powder with a known nitrate content.

5.23 pH indicator paper.

6 Apparatus

Clean all glassware thoroughly and rinse with distilled water to ensure that it is free from nitrate and nitrite ions

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg

6.2 Sample container, provided with an airtight lid.

6.3 Conical flasks, of capacity 250 ml and 500 ml.

6.4 Volumetric flasks, of nominal capacity 100 ml, 500 ml and 1 000 ml, complying with the requirements of ISO 1042, class B.

6.5 Pipettes, capable of delivering 1 ml, 2 ml, 3 ml, 6 ml, 9 ml, 10 ml, 12 ml, 20 ml, 40 ml, 50 ml and 100 ml, complying with the requirements of ISO 648, class A, or ISO 835-1. Where appropriate, burettes may be used instead of pipettes.

6.6 Graduated cylinders, of capacities 100 ml and 150 ml.

6.7 Glass beakers, of capacities 100 ml and 150 ml.

6.8 Glass funnels, with a stem of internal diameter about 2 mm.

6.9 Suitable tube, U-shaped, of length about 30 cm and internal diameter 2 mm.

6.10 Analyser, capable of determining the nitrate and nitrite contents according to the described procedure.

6.11 Suspension apparatus, appropriate to suspend the test samples.

6.12 Grinding device, appropriate for grinding the laboratory sample, if necessary.

To avoid loss of moisture the device should not produce undue heat. A hammer mill shall not be used.

6.13 Laboratory mixer or **homogenizer**, with glass containers of capacity 250 ml or 400 ml, suitable for suspending test portions of cheese and whey cheese.

6.14 Glass wool.

6.15 Spatula.

6.16 Magnetic stirrer.

6.17 Water baths, capable of being maintained at 35 °C to 40 °C and of boiling water.

6.18 Oven, capable of being maintained at 110 °C to 120 °C.

7 Sampling

Sampling is not part of the method specified in this part of ISO 14673|IDF 189. A recommended sampling method is given in ISO 707.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Store the test sample in such a way that deterioration and change in composition are prevented.

8 Preparation of test sample

8.1 Cheese

8.1.1 Prior to analysis, remove the rind or mouldy surface layer of the test sample in such a way as to obtain a sample representative of the cheese as it is usually consumed.

8.1.2 Grind the test sample by means of an appropriate device (6.12). Mix the ground mass quickly and, if possible, grind a second time and again mix thoroughly. Clean the device after grinding of each sample. If the test sample cannot be ground, mix it thoroughly by intensive stirring and kneading.

8.1.3 As soon as possible after grinding, transfer the test sample to an airtight sample container (6.2) to await the determination which, preferably, should be carried out without delay. If a delay is unavoidable, take all precautions to ensure proper conservation of the test sample and to prevent preservation of moisture on the inside surface of the container.

8.1.4 Do not examine ground cheese showing unwanted mould growth or beginning to deteriorate.

8.2 Dried milk products and infant food

Transfer the test sample into a sample container (6.2) of capacity about twice the volume of the sample. Close the container immediately. Mix the test sample thoroughly by repeatedly shaking and inverting the container.

8.3 Milk and liquid milk products

In the case of a test sample from non-skimmed milk products, adjust the temperature of the test sample to between 35 °C and 40 °C in the water bath (6.19). Mix the test sample thoroughly, but gently, by repeatedly inverting the sample bottle without causing frothing or churning, and cool quickly to approximately 20 °C.

9 Procedure

9.1 Preparation of the reduction column

9.1.1 Weigh about 10 g cadmium granules (5.2) in a glass beaker (6.7). Add about 25 ml of dilute hydrochloric acid (5.6). Wash the cadmium granules by swirling the glass beaker for several minutes. Decant the hydrochloric acid solution and subsequently wash the cadmium granules with water until the washing is pH neutral. Check the pH with indicator paper (5.23).

9.1.2 Transfer about 50 ml copper(II) sulfate solution (5.4) to the washed cadmium granules (9.1.1) in the glass beaker. Allow the obtained mixture to stand for 5 min while regularly swirling the beaker. Decant the copper sulfate solution and wash the cadmium granules 10 times with water. Keep the cadmium granules immersed in water.

9.1.3 Connect the glass funnels (6.8) with the tube (6.9), folded in a U-shape, and fill almost completely with water. By means of a spatula (6.15) transfer small portions of the cadmium granules through the funnel into the tube. Tap the tube to pack the column.

9.1.4 When the column is almost full, put glass wool plugs (6.14) of length about 1 cm into both ends of the tube.

9.1.5 Before use, run the sodium nitrate calibration solution (5.16) with the highest nitrate concentration through the column for 1 h.

9.2 Checking the reducing capacity of the column

Using the analyser (6 10), measure sequentially the absorbance of two cups containing the sodium nitrate calibration solution of 1,2 µg NO_3^- per millilitre (5 16), two cups containing water and two cups containing a sodium nitrite working solution of 0,89 µg NO_2^- per millilitre (5 18 1)

The ratio, *r*, of the average absorption of the nitrate calibration solution to the average absorption of the sodium nitrite working solution shall be at least 0,95

If the ratio, *r*, is lower than 0,95, prepare a new column

9.3 Determination of nitrate content

9.3.1 Preparation of test portion

9.3.1.1 Cheese

Weigh, to the nearest 5 mg, 2,5 g of test sample (8 1) in a glass beaker (6 7) Add 50 ml of water at about 50 °C and suspend the test portion with the aid of the suspension apparatus (6 11) Immediately cool the glass beaker with the test solution. Fill a cup of the sample exchanger with the test solution, in such a way that the separated fat remains in the glass beaker

9.3.1.2 Dried milk products and infant food

Weigh, to the nearest 5 mg, 2,5 g of test sample (8 2) in a glass beaker (6 7) Add 50 ml water and suspend the test portion with the aid of the suspension apparatus (6 11) Fill a cup of the sample exchanger with the test solution

9.3.1.3 Liquid milk products

Weigh, to the nearest 10 mg, 10 g of test sample (8 3) in a 50 ml volumetric flask (6 4) Dilute to the mark with water and mix. Fill a cup of the sample exchanger with the test solution

9.3.2 Determination

9.3.2.1 Start the pump, the spectrometer, the recorder and the data-processing equipment of the analyser (6 10) Run the reagents through the analyser according to Figure 1 for 15 min

9.3.2.2 Fill the sample exchanger of the analyser in the following order start with the six cups filled with the sodium nitrate calibration solutions (5 16), placed in order of increasing nitrate content, followed by the cups containing the test portion (9 3 1 1, 9 3 1 2 or 9 3 1 3)

9.3.2.3 Start the sample exchanger of the analyser Activate the data-processing equipment according to the directions for use. When the determination is finished, remove the reduction column (9 1) from the system and flush the system without column with an alkaline detergent (5 20) for at least 15 min and subsequently with water for at least 15 min.

9.3.2.4 Then flush the reduction column with the regenerating fluid (5 21) with a flow rate of about 2 ml/min for 15 min and subsequently again with water at the same flow rate for 10 min Then close the column carefully.

9.3.3 Dilutions

If the nitrate content of the test solution is higher than the nitrate content of the highest sodium nitrate calibration solution, repeat the analysis in duplicate by again preparing two new test solutions according to 9 3 1

Dilute the new test solutions in such a way that the expected results given by the recorder will be between the result of the lowest-but-one sodium nitrate calibration solution and that of the highest calibration solution. Then transfer the thus-diluted test solution to a cup.

9.3.4 Reference sample

Examine the reference test sample (5.22) at least in duplicate, according to 9.3.2.1 to 9.3.2.4.

9.4 Determination of nitrite content

9.4.1 Preparation of test portion

9.4.1.1 Cheese

Weigh, to the nearest 5 mg, 2,5 g of the test sample (8.1) in a glass beaker (6.7). Add 50 ml of ammonium and sodium chloride solution III (5.11), at about 50 °C, and suspend the test portion with the aid of the suspension apparatus (6.11). Immediately cool the glass beaker with the test solution. Fill a cup of the sample exchanger with the test solution, in such a way that the separated fat remains in the glass beaker.

9.4.1.2 Dried milk products and infant foods

Weigh, to the nearest 5 mg, 2,5 g of the test sample (8.2) in a glass beaker (6.7). Add 50 ml of ammonium and sodium chloride solution III (5.11) and suspend the test portion with the aid of the suspension apparatus (6.11). Fill a cup of the sample exchanger with the test solution.

9.4.1.3 Milk and liquid milk products

Weigh, to the nearest 10 mg, 10 g of the test sample (8.3) in a 50 ml volumetric flask (6.4). Make up to the mark with ammonium and sodium chloride solution III (5.11) and mix. Fill a cup of the sample exchanger with the test solution.

9.4.2 Determination

9.4.2.1 Start the pump, the spectrometer, the recorder and the data-processing equipment of the analyser (6.10). Run the reagents through the analyser according to Figure 2 for 15 min.

9.4.2.2 Fill the sample exchanger of the analyser in the following order: start with the five tubes filled with sodium nitrite calibration solutions (5.19), placed in order of increasing nitrite content, followed by the tubes containing the test solution (9.4.1.1, 9.4.1.2 or 9.4.1.3).

9.4.2.3 Start the sample exchanger of the analyser. Activate the data-processing equipment according to the directions for use. When the determination is finished, flush the system with an alkaline detergent (5.20) for at least 15 min and subsequently with water for at least 15 min.

9.4.3 Dilutions

If the nitrite content of the test solution is higher than the nitrite content of the highest sodium nitrite calibration solution, repeat the analysis in duplicate by again preparing two new test solutions according to 9.3.1.

Dilute the new test solutions in such a way that the expected results given by the recorder will be between the result of the lowest-but-one sodium nitrite calibration solution and that of the highest calibration solution. Then transfer the thus-diluted test solutions to a cup.

9.4.4 Reference test sample

Examine the reference test sample (5.22) at least in duplicate, according to 9.4.2.1 to 9.4.2.3.

9.5 Calibration curve

Calculate the most suitable calibration curve and the correlation coefficient for both the sodium nitrate and the sodium nitrite calibration solutions The obtained correlation coefficient shall be greater than 0,998 5

9.6 Checking the drift of calibration solutions

Regularly check the drift using the appropriate calibration solution mentioned in 9 2 The relative difference between two successive solutions shall not exceed 10 %. The drift path between the highest and lowest results obtained on one day shall not exceed 20 %. If these values are exceeded, check the cause and repeat the examinations.

10 Calculation and expression of results

10.1 Calculation

Convert the results given by the recorder for the test solution to micrograms of nitrate or nitrite ions per millilitre, read from the calibration curve.

Calculate the nitrate or nitrite content of the test sample by using one of the following equations

a) For dried milk products:

$$\omega_n = \omega_t \times d \times f_d$$

b) For liquid milk products:

$$\omega_n = \omega_t \times 5 \times f_l$$

c) For cheese:

$$\omega_n = \omega_t \times 20{,}5 \times f_c$$

where

ω_n is the numerical value of the nitrate or nitrite content of the test sample, in milligrams per kilogram,

ω_t is the numerical value of the nitrate or nitrite content of the test solution read from the calibration curve, in micrograms per millilitre;

d is the numerical value of the dilution factor,

f_d is the multiplying factor for dried milk and infant food obtained by the following formula (f_d = 20,6)

$$f_d = \frac{50 \times 1{,}03 \times 400}{1000}$$

f_l is the multiplying factor for liquid milk products obtained by the following formula (f_l = 5)

$$f_l = \frac{50 \times 100}{1000}$$

f_c is the multiplying factor for cheese obtained by the following formula (f_c = 20,5)

$$f_c = \frac{50 \times 1{,}025 \times 400}{1000}$$

where

50	is the conversion value to change the expression from milligrams per millilitre to milligrams per 50 ml;
1,03	is the ratio of the volume of the test solution (9.3.1.2 or 9.4.1.2) (2,5 g + 50 ml) and the volume of the calibration solutions (50 ml);
1,025	is the correction value for the moisture content of cheese (average moisture content: 50 %);
400	is the conversion value to change the expression from 2,5 g to 1 kg;
1 000	is the conversion value to change the expression from micrograms to milligrams.

10.2 Expression of results

Express the results for nitrite content to 1 decimal place and for nitrate content to whole figures.

11 Precision

11.1 General

The values for repeatability and reproducibility limits are expressed for the 95 % probability level and may not be applicable to concentration ranges and matrices other than those given.

11.2 Nitrite content

11.2.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,5 mg/kg of product.

11.2.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 1,0 mg/kg of product.

11.3 Nitrate content

11.3.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than:

— for samples with a nitrate content < 100 mg/kg:	10 mg/kg;
— for samples with a nitrate content ⩾ 100 mg/kg:	15 % of the arithmetic mean of the results.

11.3.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than:

— for samples with a nitrate content < 100 mg/kg:	15 mg/kg,
— for samples with a nitrate content ⩾ 100 mg/kg:	20 % of the arithmetic mean of the results

12 Test report

The test report shall specify:

- all information required for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this part of ISO 14673|IDF 189;
- all operating details not specified in this part of ISO 14673|IDF 189, or regarded as optional, together with details of any incident which may have influenced the result(s);
- the test result(s) obtained or, if the repeatability has been checked, the final quoted results obtained

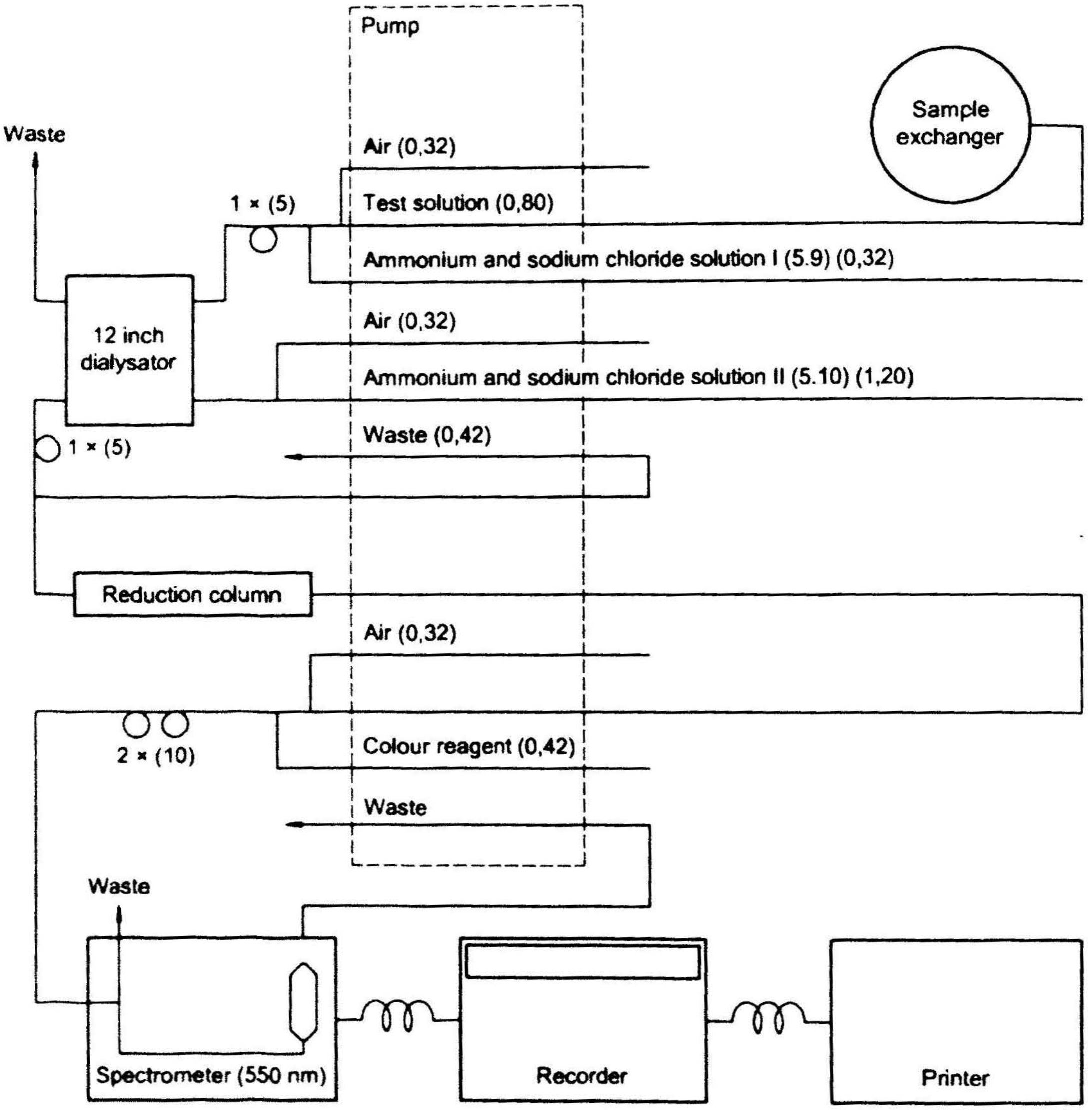

Figure 1 — Flow diagram for the determination of nitrate content

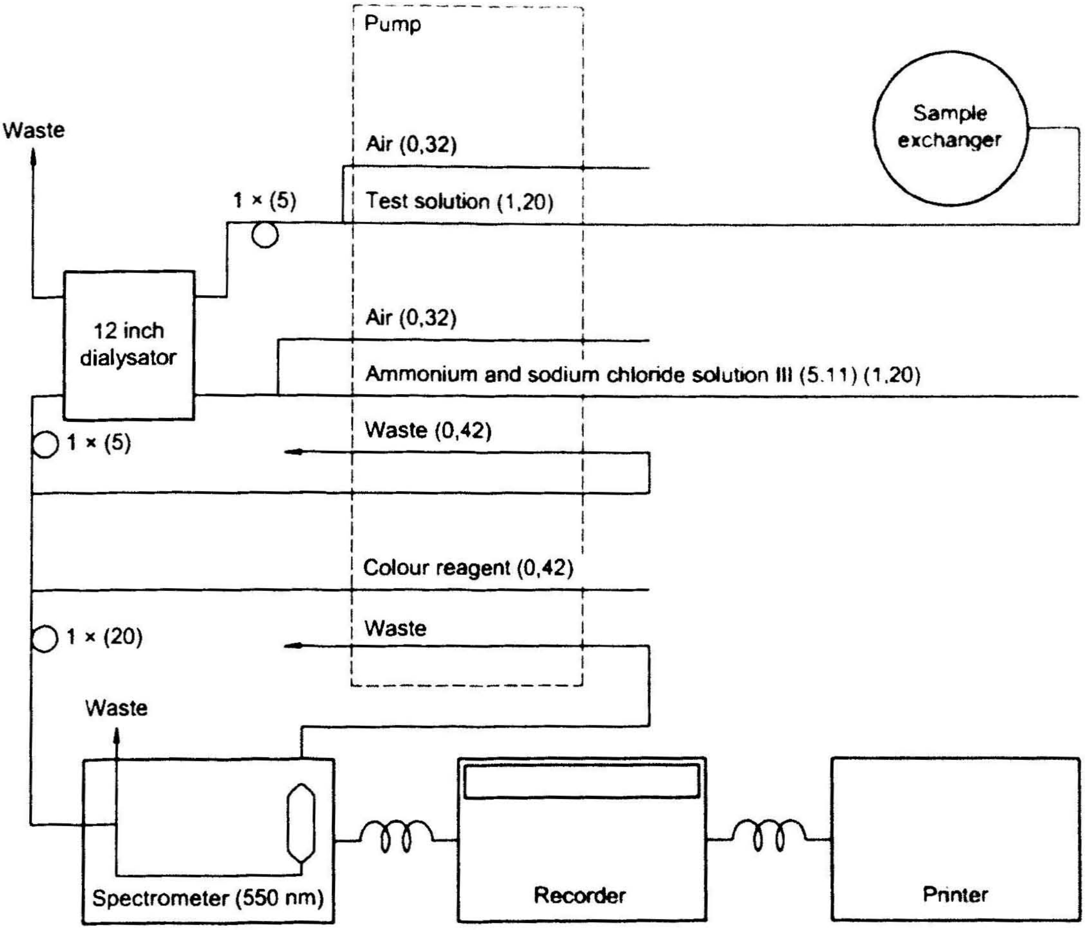

Figure 2 — Flow diagram for the determination of nitrite content

Bibliography

[1] ISO 707 1997, *Milk and milk products — Guidance on sampling*

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